IS 8634 (1987): Nitrodiazo Acid [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

SPECIFICATION FOR NITRODIAZO ACID

(First Revision)

UDC 667·281 : 547·654·3

BUREAU OF NDIANI STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

SPECIFICATION FOR NITRODIAZO ACID

(First Revision)

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Indian Standard

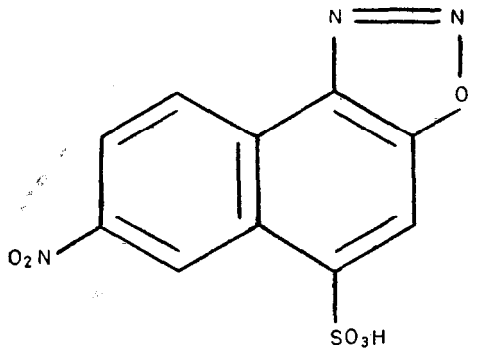
SPECIFICATION FOR NITRODIAZO ACID

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 2 January 1987, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Nitrodiazo acid ($C_{10}H_5O_6N_3S$), chemically described as 1-diazo-2-naphthol-6-nitro-4-sulphonic acid, is an important intermediate used in the manufacture of wool dyestuffs. It is obtained by nitration of diazo acid (1-diazo-2-naphthol-4-sulphonic acid). It is represented by the following structural formula:



NITRODIAZO ACID
(Molecular Mass 295·2)

0.3 This standard was first published in 1977. The Committee responsible for the preparation of this standard decided to revise it in order to modify the test methods for the requirement of free acidity and solubility in sodium hydroxide solution suitably so that the discrepancies in the earlier version are removed.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for nitrodiazo acid.

2. REQUIREMENTS

2.1 Description — The material shall be yellowish to reddish brown powder or wet cake. The material darkens on prolonged storage.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR NITRODIAZO ACID

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	55	A-1
ii)	Free acidity (as H_2SO_4), percent by mass, *Max*	15	A-2
iii)	Solubility in sodium hydroxide solution	To pass the test	A-3

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979†) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

*Rules for rounding off numerical values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*second revision*).

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number;

d) Gross, net and tare mass; and

e) Minimum cautionary notice worded as under:
'POISONOUS: AVOID INHALATION AND CONTACT WITH SKIN OR EYES.'

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by the Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics, namely, free acidity and solubility in sodium hydroxide solution shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Samples* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant/requirements given in Table 1.

*Methods of sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR NITRODIAZO ACID

A-1. ASSAY

A-1.0 Outline of the Method — The material is coupled with alkaline solution of resorcinol and resulting monoazo dye is reduced by standard titanium trichloride solution.

A-1.1 Apparatus

A-1.1.1 *Titanous Chloride Bottle*

A-1.1.2 *Reduction Flask* — 250-ml capacity, detachable from lid with ground glass joint. The lid is provided with five ground glass sockets. The central hole is for feeding titanium trichloride solution covered with kerosine while other holes are for burette, thermometer, reflux-condenser and carbon dioxide bubbling.

A-1.1.3 *Graduated Flasks*

A-1.2 Reagents

A-1.2.1 *Resorcinol Solution* — 10 percent (*m*/*v*). Dissolve 10 g of the pure substance in 40 ml of 10 percent (*m*/*v*) sodium hydroxide solution and water and dilute the solution to 100 ml with water in a volumetric flask.

*Specification for water for general laboratory use (*second revision*).

A-1.2.2 *Sodium Hydroxide Solution* — 10 percent. Dissolve 50 g of sodium hydroxide pellets in water and dilute to 500 ml in a volumetric flask.

A-1.2.3 *Dilute Acetic Acid* — 1 : 1.

A-1.2.4 *Ammonium Thiocyanate Solution* — 20 percent (*m/v*).

A-1.2.5 *Sodium Potassium Tartrate Solution* — 15 percent (*m/v*).

A-1.2.6 *Standard Titanous Chloride Solution* — 0·1 N, prepared as in **A-1.2.6.1.**

A-1.2.6.1 Prepare a 15 percent (*m/v*) solution of titanous chloride. Take 200 ml of this solution and filter through a thick pad of glasswool. Add 100 ml of concentrated hydrochloric acid and mix by passing a current of an inert gas, such as carbon dioxide or nitrogen for some time. Finally add 700 ml of boiled water and mix by passing inert gas. Store the reagent in a bottle under carbon dioxide supplied by Kipp's apparatus. Paint the bottle with black paint to protect the solution from sunlight. It is advantageous, though not necessary, to allow the reagent to stand for 10 days before it is used.

A-1.2.7 *Standard Ferric Ammonium Sulphate Solution* — 0·1 N, prepared as in **A-1.2.7.1.**

A-1.2.7.1 For preparing one litre of this solution, dissolve 58·821 g of pure ferrous ammonium sulphate $Fe[(NH_4)_2SO_4]_2.6H_2O$ in 300 ml of water and add 40 ml of concentrated sulphuric acid. Shake well. Weigh exactly 4·74 g of potassium permanganate, dissolve in 200 ml of warm water and slowly add to ferrous ammonium sulphate solution with stirring. Potassium permanganate solution should be just enough to oxidize ferrous salt. Add the last few millilitres in small portions. Cool the solution and dilute to 1 000 ml with water. Standardize the solution against a standard solution of potassium dichromate.

A-1.3 Procedure

A-1.3.1 Weigh accurately about 100 g of wet cake of nitrodiazo acid. Dissolve in water and dilute to 1 000 ml in a measuring flask. Pipette out 100 ml of the solution and couple this with 50 ml (*m/v*) resorcinol solution with 70 ml of sodium hydroxide solution at 15 to 20°C. Allow to stand for about one and a half hours. Finally dilute accurately to 1 000 ml.

A-1.3.2 Take a 10 ml aliquot portion of the solution prepared as in **A-1.3.1** into a 250-ml reduction flask, adjust the *p*H to 4 by dilute acetic acid. Add 50 ml of sodium potassium tartrate solution. Pass carbon dioxide gas in the storage bottle containing titanous chloride solution. Immediately draw 25 ml titanous chloride solution through an automatic dispenser, burette or by pipette and add it to the reduction flask. Heat

the flask in a boiling water-bath, while bubbling carbon dioxide through the solution. Titrate the excess titanous chloride against standard ferric ammonium sulphate solution at 15° to 20°C with 5 ml of ammonium thiocyanate solution added near the end point as an indicator. The end point is marked by an orange colour. Call this as reading *A*.

A-1.3.3 Determine blank reading following the procedure given in **A-1.3.2** with 25 ml of water, containing the same amounts of titanous chloride, hydrochloric acid and ammonium thiocyanate indicator solution. Call this as reading *B*.

A-1.4 Calculation

$$\text{Assay, percent by mass} = \frac{(B - A) \times N \, 1295{\cdot}2 \times 10}{M}$$

where

B = volume in ml of ferric ammonium sulphate titre reading with blank,

A = volume in ml of ferric ammonium sulphate titre reading with sample,

N = normality of ferric ammonium sulphate solution, and

M = mass in g of the material taken for the test.

A-2. FREE ACIDITY

A-2.1 Reagents

A-2.1.1 *Concentrated Hydrochloric Acid*

A-2.1.2 *Barium Chloride Solution* — 10 percent (*m/v*).

A-2.2 Procedure — Weigh accurately 10 g of the sample in a 250-ml beaker, dissolve in about 150 ml of water. Filter the solution into 500-ml measuring flask and adjust to the mark. Pipette out 50 ml aliquot sample into 250-ml beaker and add 50 ml of water. Add about 0·5 ml of hydrochloric acid. Warm the solution and then add 15 ml of barium chloride solution. Filter through ashless Whatman filter paper. Wash the precipitate with hot water till it is free from chloride. Dry in an oven, incinerate and weigh the white residue to constant mass.

A-2.3 Calculation

$$\text{Free acidity (as } H_2SO_4\text{), percent by mass} = \frac{m \times 98 \times 500 \times 100}{233{\cdot}4 \times 50 \, M}$$

where

m = mass in g of the precipitate, and

M = mass in g of the material taken for the test.

A-3. TEST FOR SOLUBILITY

A-3.1 Reagent

A-3.1.1 *Sodium Hydroxide Solution* — Dissolve 10 g of caustic soda pellets in water contained in 100-ml measuring flask and make up to the mark.

A-3.2 Procedure — Weigh about 15 g (on 100 percent assay) of the material and dissolve in 100 ml of sodium hydroxide solution. The sample shall be taken to have passed the test if a clear solution is obtained which, on filtration, does not leave any residue.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s(s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2